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DEPT OF CHEMISTRY S PONS ET AL. 30 JUL 86 TR-77  
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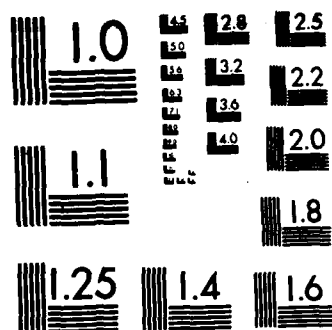
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Recent Advances in UV-Visible Reflectance Spectroelectrochemistry

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# *Trends in Analytical Chemistry*

## RECENT ADVANCES IN UV-VISIBLE REFLECTANCE SPECTROELECTROCHEMISTRY

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**ABSTRACT**

UV-Visible spectroelectrochemistry is used in situ during electrochemical experiments to examine chemical processes occurring either in solution or at the electrode surface. The technique has recently been applied to the study of polymer modified electrodes, surface adsorbed species and surface plasmons, and soluble reactive organic intermediates, in addition to many other areas of general interest. Substantial improvements have been made recently in the sensitivity of the method, which allow electrochemical kinetic and mechanistic studies on very small concentrations of electrogenerated chromophores.

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## INTRODUCTION

Conventional electrochemical methods are quite useful for kinetic studies since the rate parameters are calculated from the measured current; this quantity can be measured at extremely low magnitudes (as low as  $10^{-16}$  amperes at ultramicroelectrodes) and hence represents a very sensitive probe for investigations of reactions. However, no molecular specific information can be obtained from the data except via the value of the reversible standard potential, a quantity which may be difficult to determine in non-ideal systems or in mixed systems: the measured current represents the sum of all the electron transfer processes occurring in the system at a given potential. The field of spectroelectrochemistry was developed to provide information on the identities and kinetics of species involved in electrochemical reactions.

The first widely practiced spectroelectrochemical technique was UV-visible spectroscopy utilizing optically transparent electrodes (OTE's) [1]. The method was used to follow the electrochemical behavior of electrogenerated species in solution. Advances in UV-visible spectroelectrochemical instrumentation have increased sensitivity to the point where it is possible to investigate monolayer amounts of material on the electrode surface solution; optical studies of this type are, however, better served by reflection infrared spectroelectrochemistry [2,3].

More recently, improvements in cell design, light sources, detectors, and noise reduction techniques have broadened the usefulness of UV-visible spectroelectrochemical techniques, and presently electrochemical applications in the UV-visible region encompass a wide variety of areas. In addition to surface studies, work in this area has given mechanistic and kinetic information for a great number of organic and inorganic compounds dissolved in both aqueous and

non-aqueous solutions. The techniques have found wide use in the important field of polymer modified electrodes, which are of interest due to their possible applications in electrocatalysis and battery technology, the study of semiconductor electrochemistry, studies of the liquid-liquid interface, and investigations of ion transport in membranes. These are but a few examples of the numerous studies being undertaken.

#### SPECULAR REFLECTANCE SPECTROSCOPY: INSTRUMENTATION AND CELL DESIGN

The spectroelectrochemical technique practiced in the authors' laboratories makes use of specular reflection of radiation from an electrode polished to a mirror finish; this may typically be a platinum disk. The main advantage of this technique over transmission cells is that electrochemistry at bulk electrodes may be studied. Thus studies pertinent to important practical phenomena are undertaken with ease: the study of surface processes such as adsorption on single crystals, electrocatalysis at bulk metals, photocurrent studies on bulk semiconductors, and corrosion reactions[4].

For obtaining spectra of electrogenerated species, the detection scheme used is known as modulated specular reflectance spectroscopy (MSRS) [5,6]. The apparatus is shown in Figure 1. In this methodology UV-visible radiation from a high throughput monochromator is focussed onto the electrode, and the reflected radiation is collected and directed to a photodetector. The electrode potential is ac modulated between two potentials  $E_1$  and  $E_2$ . In a study concerned with detection of a solution soluble electrogenerated species, the base potential  $E_1$  is usually chosen such that no faradaic process occurs at the working electrode. The second potential is usually chosen so that diffusion-limited electrochemical reaction is effected, so as to generate the maximum amount of species. Since the



quantity of electrogenerated material will be modulated with the changing current (potential), the absorbance will also be ac modulated. Phase sensitive synchronous demodulation of the photodetector response leads to a signal which is proportional to the absorbance of the chromophore (vide infra). If the radiation is scanned in energy, a spectrum of the chromophore is obtained.

The cell typically used for in situ UV-visible spectroelectrochemical measurements is shown in Figure 2. The mirror disk electrode is mounted onto the end of a brass rod sheathed in an inert material such as Kel-F. The assembly is inserted into the cell through a glass barrel. Quartz windows set at a desired angle with respect to the electrode surface normal allow the radiation to pass into and after reflection pass out of the cell. A Luggin capillary reference probe is placed about 1mm from the working electrode in a position that does not obstruct the optical path. A planar secondary electrode is placed opposite and parallel to the face of the working electrode; this provides reliable linear diffusion conditions across the surface of the mirror disk for as long as ten seconds:

In order to minimize noise due to extraneous light and vibrations, the cell is mounted within a light-tight black plexiglass box atop an air-cushioned optical bench. This apparatus has been used for a number of studies including studies of chemical reactions in solution processes [5-13], surface processes [14,15], and processes occurring within polymer films deposited onto electrode surfaces [16].

The reflectance experiment has additional advantages over other in situ UV-VIS spectroelectrochemistries than that mentioned above [13]. First, the intensity of a light beam transmitted through an OTE may be attenuated considerably relative to that reflected from the corresponding solid electrode

which must result in poorer signal-to-noise ratios. Second, OTE's formed by deposition of a thin conducting film onto a transparent substrate often have very high resistances. This can lead to uneven current distribution across the surface of the OTE. Third, the reflectance technique is inherently more sensitive than the OTE technique since the light beam passes through the kinetic diffusion layer twice, thereby encountering a greater number of chromophores. Fourth, since the secondary electrode must not obstruct the light path, it is not possible in a transmission experiment to position the working electrode so that all points on its surface are equidistant from the auxiliary electrode. This leads to inhomogeneous potential distribution across the electrode surface and loss of well-defined mass transport conditions. The mathematics of mass transport has been developed only for a very few number of mass transport geometries.

Enhanced signal-to-noise ratios have recently been achieved through the use of better signal averaging techniques, more sensitive phase-sensitive detection, Fourier transform techniques, optical ac polarographic analysis, and more stable high intensity arc lamp sources. Optimized signal averaging utilizing a computer interfaced with a digital oscilloscope [10,11] has made it possible for very short transients to be obtained for species with low extinction coefficients. Arc lamps utilizing optical feedback through a photodiode have enhanced throughput and stability by increasing intensity and reducing flicker. Fiber optics have been used for both detection and as sources, and work in this area continues. Lasers have also proven to be very useful in many cases. With the incorporation of photodiode array detectors and charge coupled devices (CCD), spectral and transient data (time resolved spectra) can be obtained simultaneously [12], and the temporal behavior of several species of interest can

be followed with greater ease and speed. Sinusoidally modulated AC reflectance spectroscopy (SMACRS) [13] has increased electrochemical sensitivity manyfold by eliminating contributions to the electrochemical response due to charging current; the charging current has no inherent spectral response. Maximization of throughput and signal coupled with the minimizing of noise have made UV-visible spectroelectrochemistry one of the most powerful electroanalytical techniques.

Experiments are usually carried out in two stages. First, one obtains spectra of the species of interest by the MSRS technique. For optimum signal-to-noise ratios, the electrode potential is square wave modulated as previously described at a suitably high frequency and the amplitude of the normalized reflectance

$$\Delta R/R = (R(E_2) - R(E_1)) / R(E_1)$$

is monitored as a function of wavelength. This quantity is related to the absorbance  $A$  by the relation

$$A = \ln (\Delta R/R + 1)$$

A lock-in amplifier and frequency filter (Figure 1) are used to measure the fundamental harmonic ac reflectance signal from the photodetector tube with respect to the applied potential modulation frequency (usually 40-80 Hz). This corresponds to  $\Delta R$ . This signal must then be ratioed to the dc value of the intensity of the reflectance  $R(E_1)$ . This may be done automatically by driving the photodetector at constant gain across the spectrum, or by using an electronic divider circuit, or division with the computer controller. Absorption bands with

negative  $\Delta R/R$  values correspond to species present at the stepped potential  $E_2$ , while positive  $\Delta R/R$  bands are due to absorbing species at the base potential  $E_1$ . In this way a spectrum of reactants, intermediates, and products of a given electrode reaction sequence can be obtained.

The second stage of the experiment is to investigate the kinetics of the formation/decay of the species of interest. This is most conveniently accomplished by measuring optical transients at pertinent wavelengths. Here, the optical response to a potential step is digitally recorded. Since a single transient signal is very weak, the signal-to-noise ratio is likely to be too poor for analysis. This problem is usually overcome by signal averaging techniques. The important criterion to consider here is the recovery of the diffusion kinetic layer to relax to its original condition before recording subsequent transients. This relaxation may be expedited by automatic stirring cycles after each potential step excursion. The absorbance response recorded is related to double potential step chronocoulometry [17], and the mathematics associated with this technique can be used to describe the spectroelectrochemical response. The technique is suited both to the study of heterogeneous and homogeneous reaction rate parameters [7,8]. The specificity of the technique makes this methodology ideally suitable for resolving ambiguities in interpretation of current/potential-time measurements in these often complicated systems.

## APPLICATIONS

### SOLUTION PROCESSES

The most common application of reflectance spectroelectrochemistry is the study of electrogenerated intermediates that are soluble in the bulk solution.

An example of such a spectrum is that for one of the classic electrochemical "model" systems, the reduction of 9,10-diphenylanthracene in acetonitrile (Figure 3). As a point of interest, spectroelectrochemical data proved that the system is not simple at a platinum electrode: the reaction instead involves hydrogen which is sorbed into the platinum metal which is first reduced to hydride. The hydride ion in turn reacts with diffusing diphenylanthracene at a faster rate than heterogeneous electron transfer [5]. The spectrum at long wavelengths (negative values of absorbance) corresponds to that of the solution stable anion radical, while the band going positive at about 400nm corresponds to the spectrum of neutral diphenylanthracene, which is being consumed.

Representative transients for a system composed of a highly strained hydrocarbon and oxygen are shown in Figure 4 [10]. In the absence of oxygen (Figure 4a), the neutral molecule gives rise upon reduction to a long-lived anion radical; this transient corresponds to the formation of a stable ion radical: the couple is electrochemically reversible, and the reaction is a simple one-electron transfer. When oxygen is present in solution, the anion radical reacts with  $O_2$ , giving rise to the transient seen in Figure 4b. Notice the rapid fall-off of absorbance at the end of the applied potential pulse. Here transient spectroelectrochemical data gives mechanistic information which is unavailable from conventional voltammetric experiments.

In a similar study [11] the rate constant for the decay of a quasi-stable cation radical of a strained hydrocarbon was computed using a transient technique. At short times the cation radical is formed at a diffusion-limited rate. At long enough times, however, cation reaction occurs due to a chemical side reaction, and the expected absorbance due to the radical is less than would be expected. Figure 5 shows plots of  $-\Delta R/R$  vs.  $t^{1/2}$  for this system. From this

data the rate constant for the decay of the radical intermediate (assuming first-order or pseudo-first order kinetics) was computed from [9]

$$k = \frac{[-\Delta R/R]_0 - [\Delta R/R]_k}{\int_0^t [-\Delta R/R]_k dt}$$

where  $[-\Delta R/R]_0$  is the change in reflectance which would be obtained at time  $t$  if the absorbing species did not decay, and  $[-\Delta R/R]_k$  is the change in reflectance where decay occurs with a first order rate constant  $k$ . In this way, kinetic data for a homogeneous process involving a reactive intermediate can be easily obtained, while such information is oftentimes difficult to abstract from voltammetric data, especially for fast processes.

#### SURFACE PROCESSES

Ellipsometry is a routinely used technique for the study of films on electrode surfaces. For the electrochemist, however, this method is expensive and slow, and has consequently been used primarily for the obtaining of optical constants of films on electrode surfaces. As a result, techniques based on the monitoring of the electroreflectance effect at the electrode-solution interface have been developed. A polarized monochromatic beam is focussed on the electrode surface at a known angle of incidence and the intensity of the reflected beam is recorded as a function of wavelength, angle, potential, or time. The reflectivity of the metal will change as a function of potential due to the change in the population of conduction electrons in the surface of the electrode. The data are then compared to model calculations based on Fresnel's equations using the same three layer optical model that is used in ellipsometry, i.e. the electrode/interfacial region, bulk solution.

Recently, reflectance methods have been used to examine underpotential deposition of copper onto platinum [14]. The dependence of  $\Delta R/R$  on the coverage of adsorbed Cu atoms on a Pt crystal was examined as a function of crystal surface {(100) vs. (111) vs. (110)}. The results suggested that adsorption occurs preferentially along the (110) face, and the workers presented microspecific information on the preferential locations of adsorbed atoms as a function of coverage. This study clearly demonstrated the utility of reflectance experiments to detailed surface analysis.

The technique has been used to study surface plasmons [15]. Surface plasmons are longitudinal electromagnetic waves that propagate at the interface between a metal and a dielectric. Internally reflected radiation may be used to induce the plasmon at a precise angle of incidence. A laser source is usually used and the angle of incidence scanned until a sharp decrease in reflectivity is observed. This method has been used to study double layer effects and adsorption. Surface plasmon spectroscopy is currently being extended to investigate semiconductors and polymer modified electrode surfaces in addition to surfaces of metals.

#### POLYMER MODIFIED ELECTRODES

UV-visible spectroelectrochemistry is ideally suited to the study of polymer modified electrodes. In a recent study [16] MSRS spectra were recorded for a poly-paraphenylene film as a function of applied potential. The film was electrodeposited onto a platinum mirror electrode from the electrochemically induced polymerization of biphenyl. The spectrum of the film was then taken as a function of degree of oxidation (Figure 6). It is seen that the signal increases

with potential, and indicates the generation of two absorbing states on oxidation. The band at 620nm may be due to an excitation from the valence band to a polaron level, while the band at 380nm is attributed to band gap excitation.

Transient data for a polypyrrole film [18] on platinum film revealed that the electropolymerization reaction leading to the formation of the film was not diffusion limited. These data suggest a radical coupling reaction controlling the electropolymerization. Chronoabsorptometric data gave estimates of diffusion coefficients within the films, and also provided information on film structure.



### CONCLUDING REMARKS AND FUTURE DEVELOPMENTS

We have recently undertaken studies which involve the coupling of spectral and transient UV-VIS reflection techniques with conventional electrochemical methods such as complex plane impedance analysis and FTIR spectroelectrochemistry. New information regarding the dynamical and structural character of the electrochemical interface can be obtained from this data. Surface imaging via Raman scattering and high resolution FTIR microscopy will provide correlations between reaction rates and surface morphology. UV-visible spectroelectrochemical methods continue to be used routinely for the study of solution electrode processes, and is an important tool for the determination of mechanistic and kinetic aspects of electron transfer reactions. Work has been undertaken on the application of the technique to reactions at microelectrode arrays [19]: results provide information regarding reactions not restricted to mass transfer effects, i.e. steady state measurements under steady state conditions

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# FIGURE LEGENDS

Figure 1. MSRS experimental arrangement.

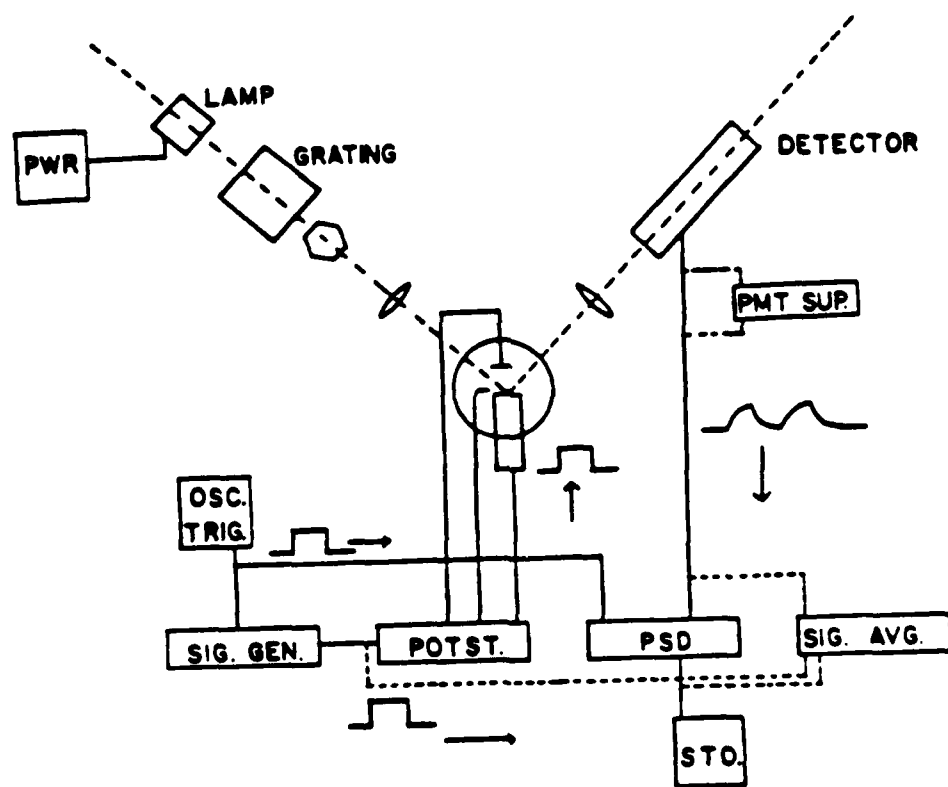
Figure 2. MSRS kinetic cell.

Figure 3. (a) MSRS spectrum of 1.0mM 9,10-diphenylanthracene at a vitreous carbon electrode (in acetonitrile, tetrabutylammonium tetrafluoroborate (0.1M), modulation frequency = 40Hz, step 0.00 to -2.50V vs. Ag/Ag<sup>+</sup>).  
(b) Same spectrum made at a platinum mirror electrode.

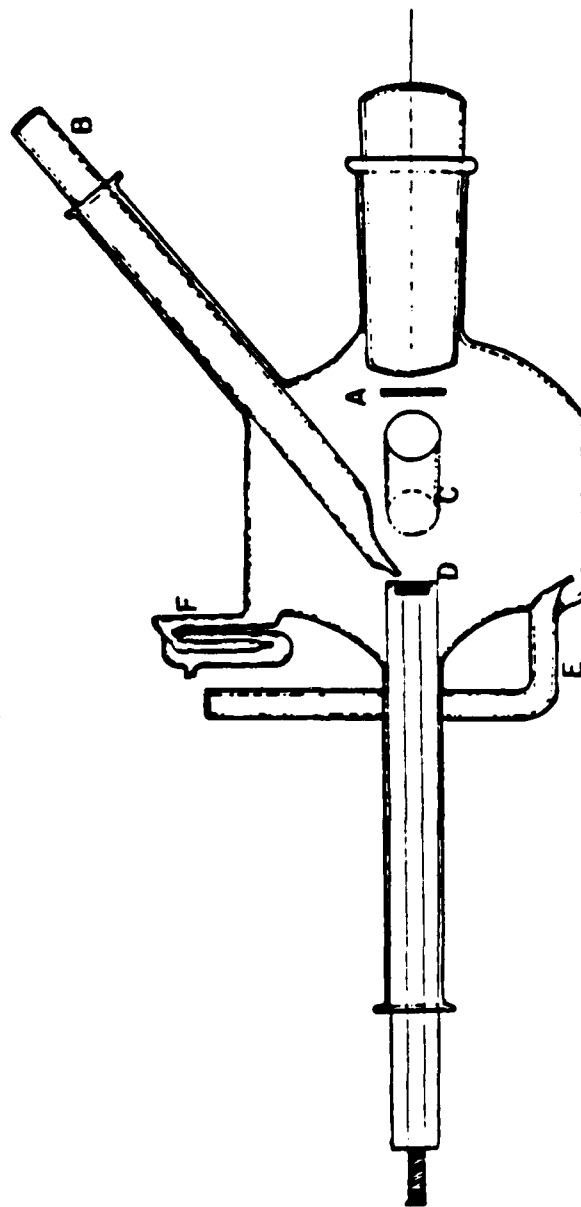
Figure 4. (a) Reflectance spectroscopic transient for the absorbance of the anion radical of 1-diphenyl methylene cyclopropa(b)napthalene (1mM in acetonitrile, 0.1M tetrabutylammonium tetrafluoroborate, potential step -1.00 to -2.10 V vs. Ag/Ag<sup>+</sup>,  $\lambda=519\text{nm}$ ). The solution is oxygen free.  
(b) Same parameters as (a), except that oxygen is present.

Figure 5. Reflectance vs. square root of time plot for the formation of the cation radical of 1,1-diphenylmethylenecyclopropabenzene from transients of duration: -20ms; 40ms; 100ms; x 200ms; o 400ms. Solution is 1mM in hydrocarbon in acetonitrile, 1M TBAF,  $\lambda=473\text{nm}$ . Potential step 0.00 to +0.90V vs. Ag/Ag<sup>+</sup>.

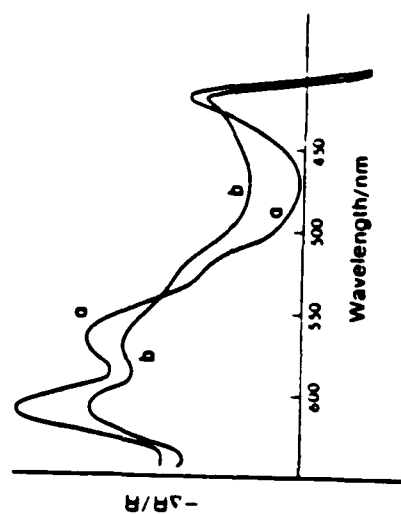
Figure 6. MSRS spectra of poly-paraphenylene film grown potentiostatically at 1.3V vs. Ag/Ag<sup>+</sup>. The applied potential was (a) 0-200mV (b) 200-400mV and (c) 400-600mV at a frequency of 80Hz.

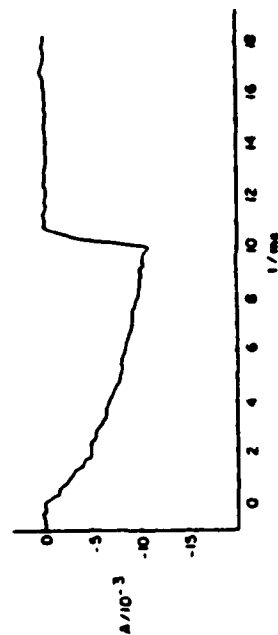
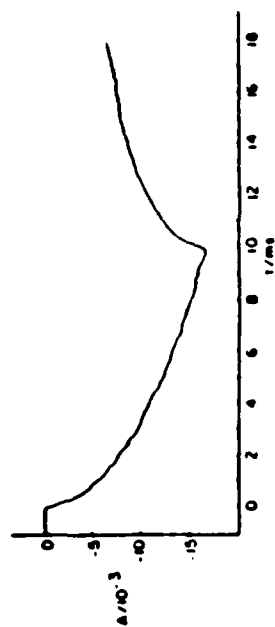


- A- Secondary Electrode
- B- Reference Electrode
- C- Light Entry(Exit) Port
- D- Working Electrode Mirror
- E- N<sub>2</sub> Entry
- F- N<sub>2</sub> Out

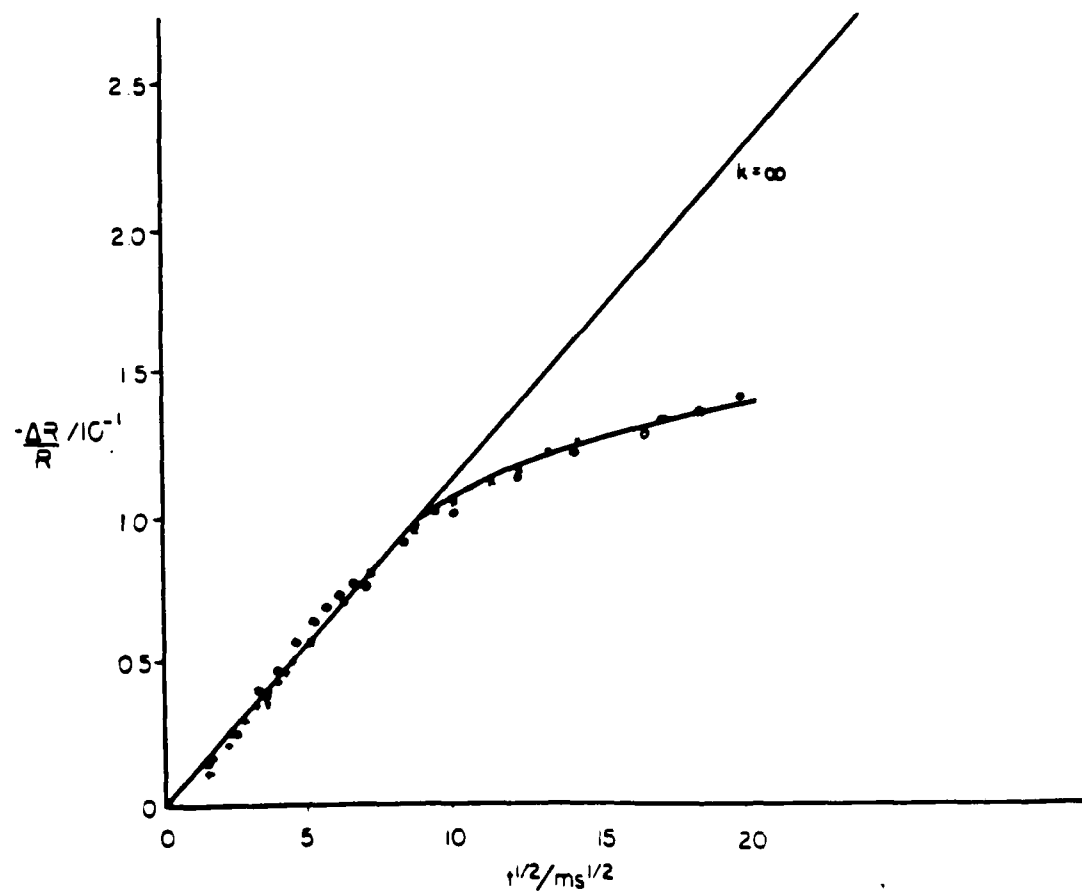


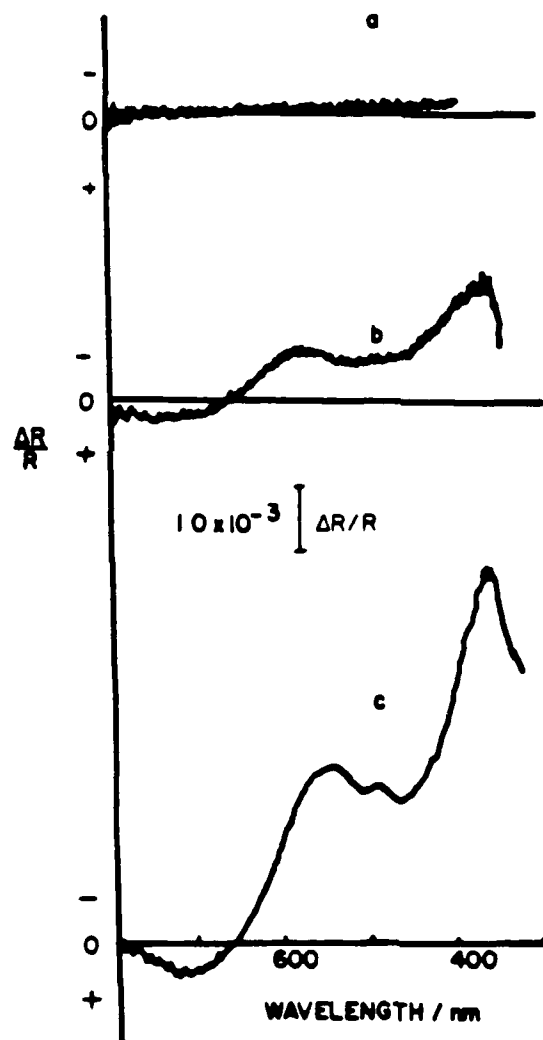
The optical cell configuration used for modulated reflectance experiments











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